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INFLUENCE OF HIGH HYDROSTATIC PRESSURE EXTRUSION ON MECHANICAL BEHAVIOR OF MATERIALS

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13. ABSTRACT

Hydrostatic extrusion is a promising new metal working process. It may provide a practical technique to form useful wrought products from materials difficult or impossible to work by other processes, and it offers the means to develope superior useful properties. This program of research is concerned with the response of a variety of materials to hydrostatic extrusion. A major objective is to relate the microstructure and mechanical properties of extruded materials to the important processing variables, extrusion ratio, extrusion pressure, temperature, and extrusion rate. The experiments fall into two categories; 1. A detailed, systematic investigation of the influence of the processing variables on the properties of several fairly simple materials (pure Fe, Ni, Mg, and Ti; and Cu-30Zn or Cu-10Sn) 2. The utilization of hydrostatic extrusion to optimize the properties of more complex materials chosen for their technological utility (Mg-Li-B for high specific stiffness; Fe-C for high strength, ductility; T-D Nichrome for high temperature strength; Al-Fe, a high conductivity, low density material; NbTi a super conducting alloy). The theoretical portion of the program is devoted to an understanding of extrusion. Elastic-plastic finite-element programs are being considered as a means of analyzing the extrusion process. Such studies are directed towards a complete stress analysis of extrusion and should lead to a better elucidation of the factors influencing the onset of undesirable defects during extrusion.

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extrusion rate						
extrusion ratio						

10

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1. Introduction

Hydrostatic extrusion is the metal working operation that consists

of forcing metal through a die as in conventional extrusion but with the

addition of a superimposed hydrostatic pressure. The effect of the super-

imposed hydrostatic pressure is to permit the extrusion of materials not

practically extrudable with the conventional process, it allows greater

extrusion ratios to be used, and it seems to impart improved mechanical

properties. This promising metal forming technique has already had some

technological applications, and its potential for industrial utilization

is very attractive.

In view of the considerable technological interest in hydrostatic

extrusion it is surprising how little information has been gained about

the basic engineering mechanics of the process or about how the variables

of the process relate to the resulting mechanical properties of hydro-

statically extruded materials. It is the intent of the present program

to make inroads on both these deficiencies.

2. Hydrostatic Extrusion Press

A hydrostatic extrusion press is being built for this work by Revere

Copper and Brass Corp. Funds for the purchase of this device have been

provided by an equipment grant from the National Science Foundation and

by Stanford University. Delivery date for the machine has now been set

at February 20, 1975.

The apparatus under construction is only the second hydrostatic press

1.

of this basic design to be built. We initiated a major design change which will permit essentially complete control of the extrusion variables, a factor crucial to material preparation and to the testing of theoretical work. This design alteration operates in the direct extrusion mode and will be discussed below. The press will be capable of applying a maximum pressure of 500,000 psi during the extrusion of a 3/8 inch diameter billet or 250,000 psi with a 3/4 inch diameter billet. The extrusion rate is controllable from 0 to 72 inches per minute (relative speed between die and billet). This range of extrusion rates will enable us to control heating from essentially isothermal conditions to fast rates where adiabatic heating of hundreds of degrees will occur. Most of the planned work will be done with the material and apparatus initially at room temperature. However, the device is designed to permit hot extrusion up to 600°F and possibly higher. In addition to the above features the machine may be operated in either indirect (back) extrusion or direct extrusion modes. Operation of these two extrusion modes, (Figures 1 and 2, respectively), will be briefly described below.

As shown in Figure 1, a billet and high pressure fluid are worked into a work chamber. As the pressure intensifier piston pushes the die against the billet, a seal is formed. Further movement of the piston causes a hydrostatic pressure to build up in the work chamber. The pressure will continue to rise until the pressure differential between work chamber and outlet chamber is sufficient to extrude the material, at which time steady state conditions are reached. Therefore, one can see that the work chamber pressure (with this extrusion mode) is a function of extrusion rate, material properties, and die reduction ratios. Although the extrusion cannot be
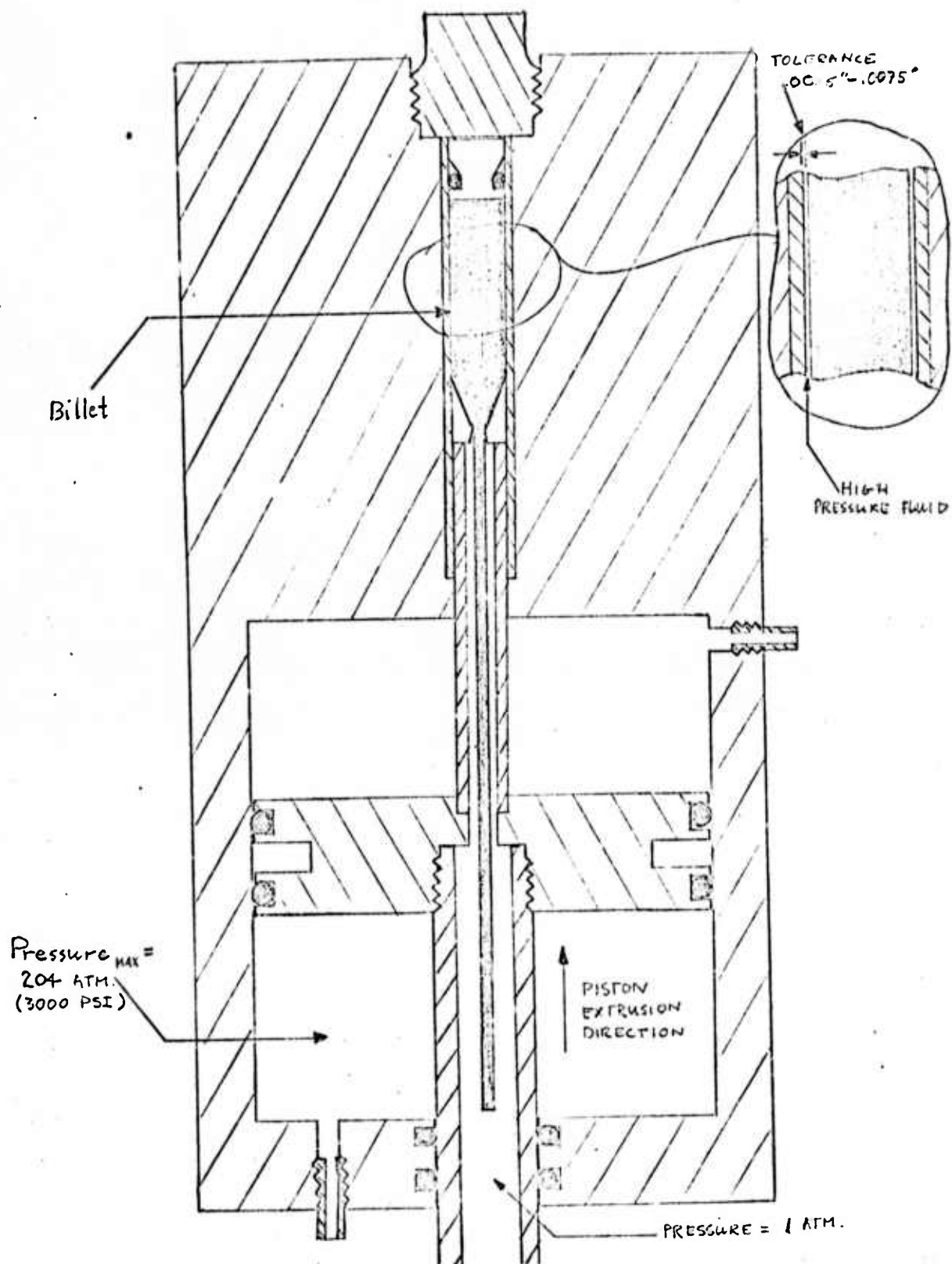
Figure 1. Machine design for operation in indirect or back extrusion mode.

considered as "truly hydrostatic" since the outlet chamber pressure is one atmosphere, mechanical tests of such extruded materials have revealed impressive properties. It would be very useful, however, to have a controllable pressurized outlet chamber.

For this reason we modified the Revere model to conform to the design as shown in Figure 2. In this mode a plunger pushes the billet through a stationary die. As before, the hydrostatic pressure in the work chamber increases as force is applied to the billet until the differential pressure is large enough to extrude the material. Now, however, the material is not extruded into a one atmosphere outlet chamber, but into an independently controllable hydrostatic pressure. This then is "true hydrostatic extrusion" in the sense that the whole process is under hydrostatic pressure. Besides the obvious advantage of having the complete extrusion under high pressure, control of outlet chamber pressure means that the work chamber pressure is no longer only a function of extrusion rate, material properties, and die reduction ratio. This control of superimposed hydrostatic pressure will permit experiments to be performed that will differentiate between the true effect of pressure (the Bridgman effect) and the effect of the elimination of friction in the die and in the billet chamber.

3. Laboratory Space

An area of 800 feet has been arranged for the installation of the hydrostatic extrusion press and its attendant tooling and gear in the Materials Testing Laboratory adjoining the Materials Science and Engineering Department at Stanford University. This will be most convenient because this same laboratory houses the mechanical test equipment to be used for measuring the mechanical properties of the extruded materials and because
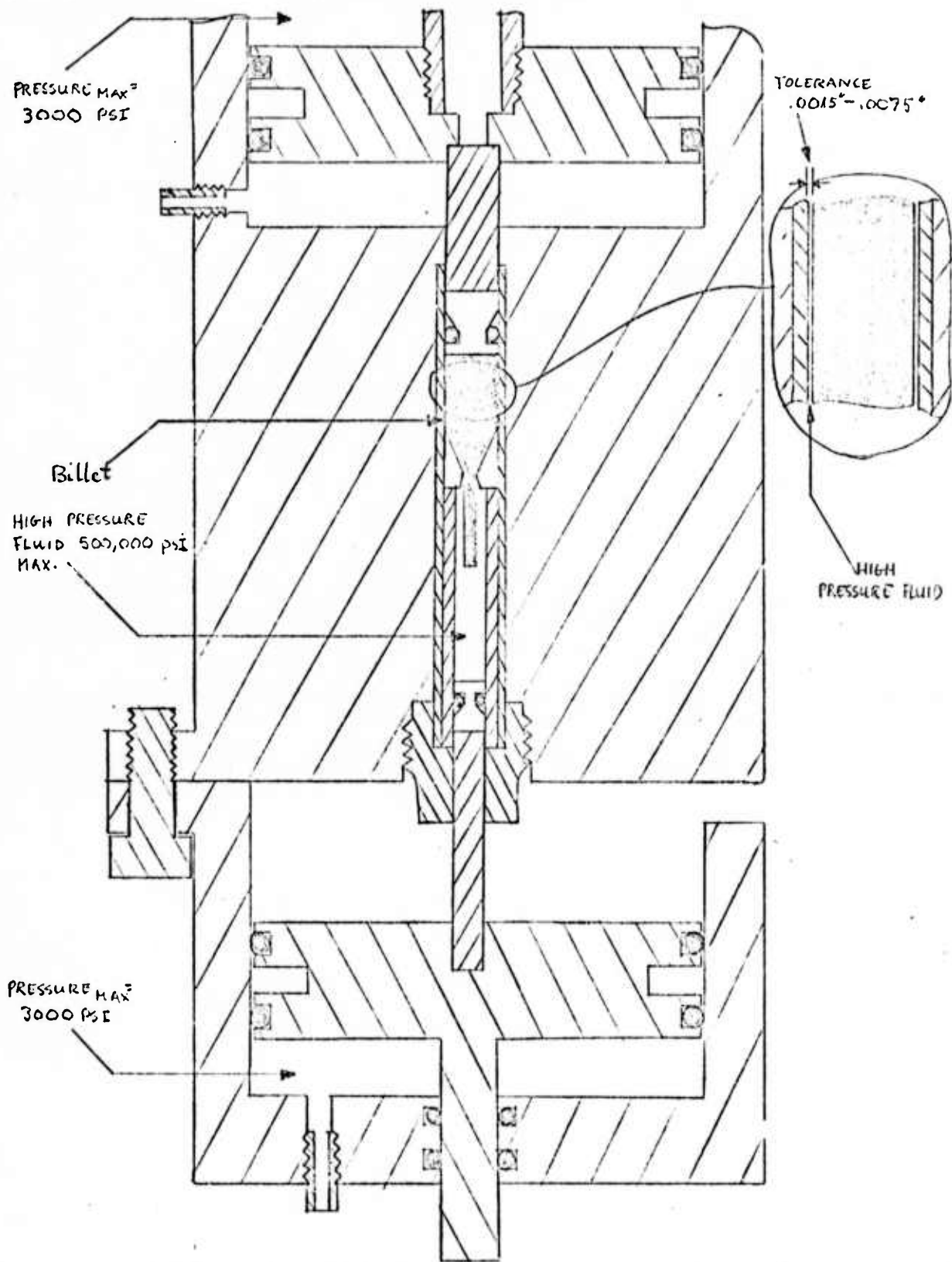
Figure 2.   Machine design for operation in direct extrusion mode.   "True hydrostatic pressure".

the Materials Science departmental machine shop is immediately adjacent.

4. Testing Equipment and Materials

Mechanical property measurements will be an important part of this program. It is planned that both tension and compression tests will be performed on hydrostatically extruded experimental materials. For that purpose a fully instrumented, 20,000 pound capacity, hydraulic mechanical testing system (MTS model No. 901.29, Research Inc.) has been set up adjacent to the location for the hydrostatic extrusion press. This mechanical test equipment is now fully operational.

We are studying the details of the mechanical testing techniques which we will be using. The small size of the extruded rod materials necessitates some special tooling and sample preparation methods which are currently being improvised.

One of the first materials which we plan to extrude hydrostatically is pure aluminum containing a small amount of iron (.05 to 1 weight percent). Previous work in this department has revealed that the creep resistance of such a material can be increased enormously by the presence of subgrains stabilized by small amounts of iron in the form of the intermetallic compound $FeAl_3$. We have successfully prepared rods of aluminum-iron in single crystalline form in order to eliminate the detrimental contribution of grain boundaries to creep; the resulting rods will be extruded hydrostatically at room temperature and under isothermal conditions in order to develop a fine subgrain structure. Its mechanical and electrical properties will then be evaluated and correlated with the corresponding microstructure. Such a material could have great promise as electrical conduction wire with high creep resistance.

In our original hydrostatic extrusion proposal we indicated that we would be studying the mechanical behavior of particulate composites. Such composites are oftentimes difficult to extrude by ordinary methods and should be ideally suited for testing the feasibility of hydrostatic extrusion. Earlier work by us in the preparation of particulate composites involved the use of a special powder metallurgy-mechanical comminution process[1,2] developed at Stanford. This work lead to considerable success in obtaining unusual mechanical properties in large volume fraction particulate composites based on model systems (zinc and cadmium base metals containing alumina, tungsten and boron)[3-6]. Recently we have concentrated on magnesium and copper base particulate composites. These systems have been more difficult to study; in great part this is because our PMMC process was not as successful, in many instances, in giving us adequately uniform distributions of particles in the higher melting matrix materials we had chosen. Two sources of difficulties were encountered: (1) in our mixing operation prior to sintering we dealt with coarse matrix powders (in the order of 30-40 microns) and fine second phase powders (in the order of 1 micron). These large size differences lead to difficulty in obtaining a uniform distribution of hard particles in a given matrix material. (2) Our pressure sintering and extrusion operation was limited to low pressures, about 30,000 psi, for the size billets used. Such pressures were oftentimes too low to obtain high density billets during sintering leading to many voids. Furthermore, inadequate oxidation protection during sintering resulted in products which oftentimes could not be sucessfully extruded even with heavy duty presses (a 300 ton capacity extrusion press was used at Lockheed Research Laboratory in Palo Alto).

As part of our hydrostatic extrusion program we propose to prepare

7.

particulate composites of copper containing alumina in such a way that highly uniform distributions of the second phase will be achieved with one hundred percent densification. For this purpose we have purchased a mechanical attritor from the Union Process Co. of Akron, Ohio. The attritor's principal quality is that it can not only throughout mix powder but it crushes them at the same time. The powders are reduced in size continuously during mechanical comminution. Thus, typical powders of copper, initially in the order of 30-40 microns, will be reduced in size to one micron which is in the same order of size as the alumina additions. The mechanical comminution will be performed in a protective atmosphere, a special feature of the attritor we are obtaining. Having the same size particles for both phases should lead to a sintered product with reasonably uniform distribution of the second phase in the matrix material. Sintering at high temperature will be performed in the Lockheed compression press (200 ton capacity) which will increase our sintering capacity by a factor of four, to 120,000 psi. A protective atmosphere will be provided with the system we are now preparing and the new pressure sintering arrangement should yield one hundred percent dense compacts with a minimum of contamination. We will then plan to use our hydrostatic extrusion press to obtain the desired degree of mechanical working with the end objective of evaluating the mechanical properties of such particulate composites. Optimum dispersions as obtained previously with our zinc and cadmium composites[1-6] should result.

Mr. Robert Whalen, a Research Associate and Mr. Robert Caligiuri, a graduate Research Assistant, are actively engaged in all experimental phases of our hydrostatic extrusion program.

5. Development towards the Analysis of the Extrusion Process

The analytical portion of our hydrostatic extrusion program is being carried out principally by Professors E. H. Lee and R. L. Mallett of the

Applied Mechanics Department. In the following sections the limitations of plastic-rigid analysis of metal forming problems are discussed in order to illustrate the need to base evaluation of process design variables and material properties limitations on elastic-plastic analysis. For example, the onset of extrusion defects, which limit satisfactory process operation, commonly depends on a complete stress analysis which is provided only by elastic-plastic theory. Elastic-plastic finite-element programs are discussed especially as they relate to the analysis of the extrusion process. Modifications needed to meet the requirements of steady-state analysis are examined. To date the steady-state problem has not been analyzed although it governs extrusion operations in practice. Plausible approaches to the steady-state problem are discussed.

a. Introduction

In many metal forming processes plastic deformation occurs non-homogeneously on the scale of the region undergoing deformation due to the geometry of the dies or container and to friction between the flowing metal and the supporting structure. Thus in extrusion through a conical die, the sudden change in direction of the material velocity when the billet enters the die and again when it leaves, causes a complicated local deformation pattern and this is enhanced by the retarding effect of friction at the die surfaces. If one wishes to abstract the influence of a particular metallurgical characteristic which will generally have an effect on a much more local scale, it would clearly be advantageous to be able to analyze the macroscopic deformation pattern and so to assess the influences of the metallurgical factor when associated with a known strain history. Without a macroscopic analysis, one would have to try to make deductions from properties of the process as a whole. These properties comprise averages

9.

over a spacial strain distribution which could be far from uniform. We therefore decided to attempt to develop the capability of analyzing the stress and strain distributions during extrusion, so that these could be evaluated for different material characteristics, die angles and shapes, extension ratios and friction conditions.

Comparison with experiments using the hydrostatic extrusion press will hopefully lead to refinement and validation of the theory, which can then be used to predict the onset of extrusion defects, such as the generation of internal cracks, and how these are influenced by material characteristics and metallurgical factors. In effect a macroscopic analysis would permit the influence of metallurgical characteristics to be assessed in terms of their effect on stress-strain relations, which express material response to homogeneous stress and strain distributions and thus exhibit the influence of material characteristics more sensitively. This is so because the response of all elements to a certain feature will occur simultaneously in contrast to a general metal forming process in which a broad range of strain exists in the specimen at each instant. A simple example of this circumstance is represented by the condition for the onset of internal cracking along the axis of symmetry. This could no doubt be expressed in terms of the stress variation along the axis, but hardly in terms of global characteristics of the process as a whole, at least in any precise manner.

b. Material Characteristics and Metal Forming Analysis

Figure 3a. illustrates a typical stress-strain curve in tension for an aluminum alloy. The initial loading along OA represents elastic response with modulus E. When the yield stress Y is exceeded, plastic flow occurs along AB. Unloading from B takes place along an elastic line BC, where C corresponds to
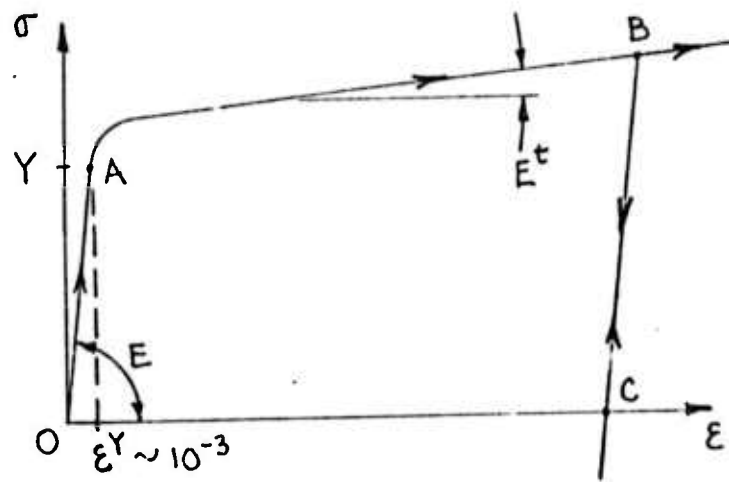
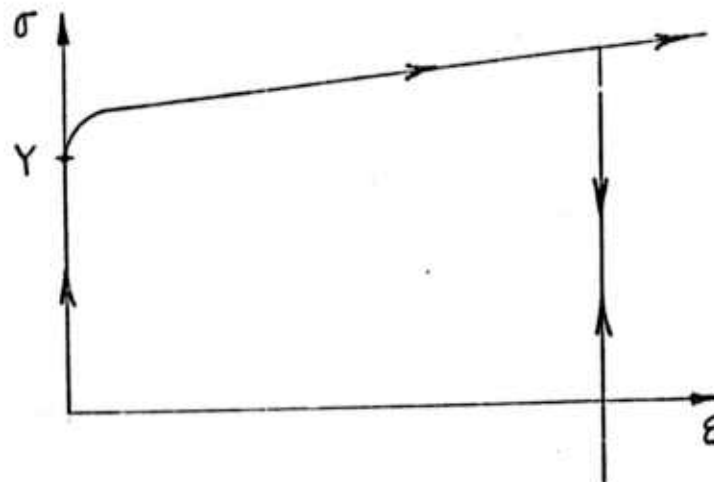Fig. 3a. Elastic-plastic stress-strain relation.

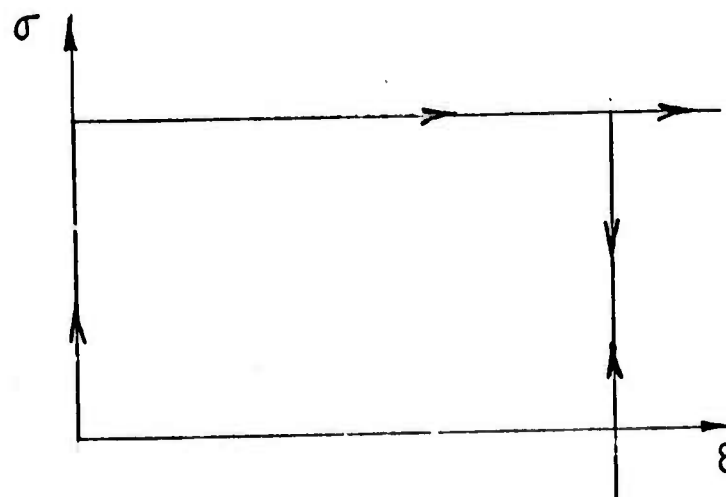
Fig. 3b. Plastic-rigid stress-strain relation.

Fig. 3 c. Ideally-plastic-rigid stress-strain relation.

11.

zero stress, so that OC represents the "permanent" or plastic strain. The total strain at B is made up of the plastic component OC, and the elastic component given by the increase in strain from C to B.

The elastic strain at yield, ε^Y, has the magnitude Y/E which is of the order 10^{-3}. Metal forming can correspond to extension of AB to strains of the order unity, so that elastic strain magnitudes ~ 10^{-3} are negligible by comparison. It might therefore seem appropriate in metal-forming analysis to neglect elastic strains and hence adopt a plastic-rigid theory for which Figure 3a is replaced by Figure 3b in which elastic strains are of zero magnitude.

In Figure 3a the strain-hardening coefficient or tangent modulus E^t is commonly small compared with Young's modulus E ($E^t/E \sim 10^{-3}$), so that it could be appropriate also to neglect E^t and hence obtain the ideally-plastic-rigid relation illustrated in Figure 3c.

In utilizing plastic-rigid theory, the material is considered to be divided into plastic zones where appreciable flow is taking place and "rigid" regions. In fact the latter constitute regions of contained plastic flow where the constraint of surrounding elastic material permits plasticity but limits strains to be of the order of elastic strains. While some metal forming solutions have been obtained on the basis of plastic-rigid theory, for general problems difficulties arise in determining the rigid regions, since, with no strain, only the equilibrium equations are available to determine the stresses and these are insufficient. The rigid regions are needed to delineate the domains of large deformation and hence to define the plastic part of the solution. Thus, in general, plastic-rigid theory cannot supply metal forming solutions. In certain cases, the slip line field of a domain flowing plastically suggest appropriate boundaries for the regions of contained plastic flow, which can be validated

12.

using the lower bound limit analysis theorem based on the existence of a safe statically admissible stress field[7]. In such cases the plastic flow solution determines the strain distribution to within a possible inaccuracy of elastic strain magnitude.

For metal forming problems for which plastic-rigid analyses can be carried out, the distribution of major plastic strain is determined and consequently the power required for the process is also determined. This includes the contribution to the so-called lost work associated with the generation of deformation which does not contribute to the objective of the process. For example, Figure 4, from Hill[8], shows the deformation pattern associated with sheet extrusion through a rough die. Initially square cells are not only compressed laterally and stretched longitudinally to achieve the reduction in section, but also the die produces shear strain by holding back material adjacent to the surfaces, and this produces no change in section. The energy expended in generating this parasitic deformation reduces the efficiency of the process, and an objective of die design is to reduce this loss. Because elastic strains are neglected in such analysis, stresses cannot be determined in regions not involving appreciable plastic flow, and this includes, for example, residual stresses in the product. Stress variation in the process has a major bearing on the development of extrusion defects such as the initiation of cracks or roughening surface deformation. It is such aspects that provide limits in die design and in material characteristics which will ensure the achievement of satisfactory extrusion. Thus elastic-plastic analysis is needed to provide a satisfactory basis for the evaluation of process variables and limitations. This calls for numerical analysis in other than the simplest problems, and the finite-element procedure appears to offer most promise. We have developed such
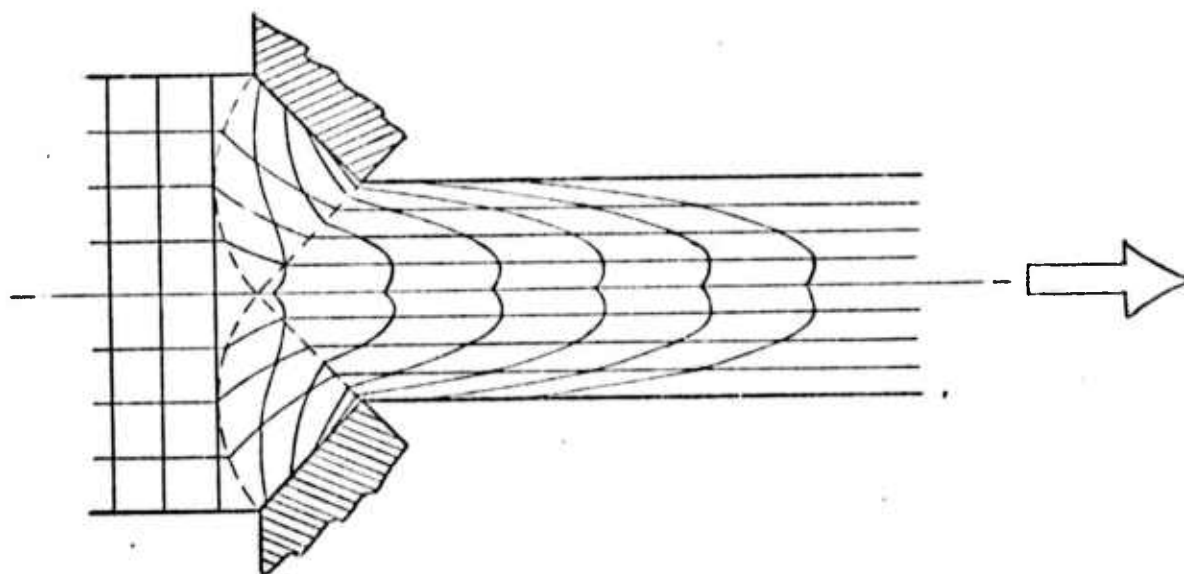
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Fig. 4. Deformation of a square grid due to sheet extrusion
through a rough die according to plastic-rigid theory.

a program and are looking into the utilization of it for the purpose of analyzing the extrusion process.

c. Elastic-Plastic Analysis

During the last year or so we have developed an elastic-plastic finite-element program to analyze metal forming problems involving finite strains[9,10]. It utilizes an isotropic work hardening law based on a measured tensile stress-strain relation. It was used to determine the macroscopic force-deformation relation for a porous metal, taking account of the collapse of the pores. Provision for inclusion of inertia terms was incorporated, but the effect of inertia can be negligible in many metal-forming problems and it is simpler not to include this provision. In accordance with the incremental nature of plasticity laws, the program develops the deformation in a step-by-step fashion by means of a sequence of strain increments. Some initial problems of numerical instability when plastic flow first sets in, due to the sudden reduction in magnitude of the tangent modulus, have been overcome.

The program mentioned above, in common with other elastic plastic programs, treats the transient problem of the developement of plastic deformation from prescribed initial conditions and boundary conditions. Such programs have been utilized in attempts to analyze the steady state extrusion problem indirectly by considering the metal which is to be extruded as initially unstrained and occupying the space in the die and in the entrance and exit regions[11,12]. Motion of the billet is then prescribed by boundary conditions, and steady state extrusion would gradually develop as the metal flows through the die. However, either the cost of carrying through the calculation until a steady state has been reached or the error build up have apparently been prohibitive in such procedures, since the computations were continued only until strains of the

15.

order of 1% had been generated. This is far from steady state as is evidenced by the fact that the driving force required continues to increase. It is the steady state which constitutes the process of extrusion in normal production runs. Thus the stress distributions calculated so far provide only very inadequate information for rational design of the process.

We are working on methods of utilizing elastic-plastic programs to attack the steady state problem directly. It turns out that close attention must be paid to the accuracy of the algorithms used in formulating such programs in order to analyze the steady-state case. This is due to the fact that many of the elastic-plastic programs in current use introduce errors because of a lack of preciseness in defining increments of stress and strain. Increments pertaining to a particular material element are needed in stress-strain relations while increments at a fixed point in space are needed in the simplest formulation of the equations of equilibrium.

Thus if $\sigma_{ij}(\underset{\sim}{x},t)$ expresses the varying stress distribution, where the coordinates $\underset{\sim}{x} = (x_1, x_2, x_3)$ give the current position and t the time, the equilibrium equations take the form:

$$\frac{\partial \sigma_{ij}}{\partial x_j}\bigg|_t = 0 \qquad (1)$$

with the usual summation convention on the repeated j. For incremental analyses, common in plasticity theory, it is convenient to express (1) in stress-rate form or stress-increment form. Writing

$$\delta(\sigma_{ij}) = \frac{\partial \sigma_{ij}}{\partial t}\bigg|_{\underset{\sim}{x}} dt \qquad (2)$$

for the change in stress at a fixed point in space during the time increment dt, equation (1) implies that either

$$\frac{\partial}{\partial \bar{x}_j} \left(\frac{\partial \sigma_{ij}}{\partial t} \right) = 0 \tag{3}$$

or

$$\frac{\partial}{\partial \bar{x}_j} \left(\delta \sigma_{ij} \right) = 0 \tag{4}$$

The increment of stress following a particle which must be used in the incremental stress-strain relation is given by the convected derivative

$$\frac{d\sigma_{ij}}{dt} = \frac{\partial \sigma_{ij}}{\partial t} + v_k \frac{\partial \sigma_{ij}}{\partial x_k} \tag{5}$$

where v_k is the material velocity. Thus the increment of stress in a material element during the time interval dt is given by:

$$d\sigma_{ij} = \delta \sigma_{ij} + du_k \frac{\partial \sigma_{ij}}{\partial x_k} \tag{6}$$

where $du_k = v_k \, dt$. In the development of finite element programs the stress-strain laws and the equilibrium equations, or equivalently the principle of virtual work, are combined and in many cases the types of increments used have not been distinguished. These questions have been discussed by Hill[8], p. 54, and recently examined by Rice[13]. It has been common to consider the second term on the right hand side of (6) to be negligible compared with the first term so that the distinction between $\delta \sigma_{ij}$ and $d\sigma_{ij}$ is lost. This is satisfactory in classical elasticity, but can lead to appreciable error in incremental plastic analysis. In particular, for steady-state, $\delta \sigma_{ij} \equiv 0$ so that the last term in (6) can clearly not be neglected. We are examining the structure of the theory with such points in mind, and hope to be able to obtain a formulation which will permit direct evaluation of the steady-state extrusion problem.

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18.